The Stability of Bredig Gold Sols

*

DISSERTATION

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN
THE FACULTY OF PURE SCIENCE IN
COLUMBIA UNIVERSITY

*

BY

PAUL BALLINGER DAVIDSON, B.S.

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DEDICATED TO

MY MOTHER AND FATHER

ACKNOWLEDGMENT

The author wishes to express to Professor H. T. Beans his sincere thanks for the suggestion of the problem and for his generous assistance and advise throughout the course of the investigation.

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The Stability of Bredig Gold Sols

Introduction

In a comprehensive study of Bredig gold sols it is necessary to established criteria of their stability. Various criteria have been offered but they are most unsatisfactory as well as indefinite and prove to be but qualitative in nature.

If gold sols were merely mechanical suspensions of metallic gold particles in water then the most logical differentiation would be that based upon size of particles as advocated by many authors, particularly Zsigmondy[1], Ostwald[2], Bancroft[3], and Burton[4]. Zsigmondy, for example, classifies a gold sol as stable when the size of its particles falls between the limits of 1μ to $1\mu\mu$.

In light of the work conducted in this laboratory such as classification or correlation of size of particles and stability has no meaning whatever. It has been clearly shown that Bredig gold sols are more complicated systems than can be represented as physical suspensions of metallic gold particles in water. If degree of dispersion were the determining factor in the stability of a sol then it should be possible to prepare stable gold sols by the Bredig method by using pure water as the dispersion medium. The gold vapor in the arc is suddenly cooled by contact with water and at first condenses into solid of very high degree of dispersion. Only after a considerable amount of gold had been dispersed in water would particles of larger dimensions occur and thus render the sol unstable. Beans and Eastlack[5] however have shown that it is not possible to prepare any thing but an unstable suspension by arcing between gold electrodes in pure water, that is water having a specific conductivity lower 10^{-6} mhos. In order that a stable sol may form, an electrolyte must be present in the solution which comes in contact with the gold vapor of the arc. Furthermore this electrolyte must furnish an anion which can serve as a nucleus of condensation for gold vapor or in some way be capable of combining with the gold. Only ions which form stable compounds with gold can be used as these "stabilizing" ions. In the face of these facts the process of formation of these stable sols is certainly not purely physical but consists of some kind of compound or complex formation. This compound does not actually form in the chemical sense, for it has been shown that no displacement of the cathion by gold occurs and the solution contains no gold ion. The compound formation is therefore of the nature of an adsorption compound between the metallic gold particle and the anion of the electrolyte. It is probable that these adsorption compounds can be formed only with those elements whose atomic structure bears the necessary relationship to that of the gold atoms to permit of chemical combination.

The work of Beans and Eastlack therefore constituted an extension of the "complex" or chemical theory of colloid formation,

which had been developed for inorganic hydrophilic sols by Linder & Picton[6], Jordes & Kauter[7], Svedberg[8], and Von Weimarn[9] to the hydrophobic sols of which gold is an example.

J. A. Wilson[10] has made a definite and significant contribution to the theory of gold sol formation by applying the Donnan membrane theory to the complex formed between the gold particle and the electrolyte which will be discussed later.

Working in this laboratory Beaver[11] has investigated the nature of the adsorption complex in Bredig gold sols; Miller[12] has studied the relationship between the electrolyte concentration and the maximum concentration of gold; and Briggs[13] has investigated the change of degree of dispersion with time of arcing of gold sols prepared in varying concentrations of one electrolyte.

With the evidence furnished by the researches noted above, it must be apparent that any attempt to interpret the stability of Bredig gold sols in terms of size of particles is fultile and without scientific meaning. Furthermore it may be noted in passing that there are no methods known for the determination of the size of gold particles in a sol. The methods proposed involve sources of error and assumptions which place them in the catagory of mere guesses rather than determinations. For example the ultramicroscopic method of Zsigmondy involves the necessity of instantaneous estimation of a large number of rapidly moving points of light and the assumption that there is a visible point of light for each gold particle within the micrometric field. It further involves the assumption that the specific gravity of the colloidal gold particle is the same as that of massive gold whereas the material obtained by centrifuging the sol bears no resemblance to metallic gold.

Returning to a consideration of some of the other criteria of stability of gold sols in common use, mention may be made of the color of the sol. Here we find the statement either definitely made or tacitly assumed that there is a relationship between the color of the sol and the size of the particles. The evidence of work done in this laboratory is that there is no such relationship since Briggs always obtained pure red sols although the degree of dispersion must have varied between wide limits. Furthermore all Bredig platinum sols are black or nearly so although their degree of dispersion is of the same order as that of the red gold sols. In this connection the following table from Zsigmondy is also illuminating.[14]

Color of gold sol	Average size in $\mu\mu$
Rose	6
Deep red	10, 15, 18, 32
Violet red	23, 32, 75
Violet	35, 95,
Blue violet	54
Dirty blue	30
Purple red	38

While it is true that upon addition of electrolyte to red gold sols the color changes to purple before precipitation occurs nevertheless some purple gold sols are as stable as those showing a red color.

To state that a sol is stable if the size of its particles falls between certain limits, or that it will not precipitate out after standing for indefinite periods of time, or that its color is red, violet or purple leaves one with a very indefinite idea as to just what stability is. It is often alleged that stability is determined by the amount of added electrolyte necessary to precipitate the sol but here the rate of adding the electrolyte and the nature of the precipitating ions causes unreliable results. If the Hofmeister series held true in all cases and local concentration effects could be overcome this might be of some use. Stability then resolves itself into gravitational effects which can be increased and controlled by centrifugal methods. The author offers a more reliable criterion of stability of Bredig sols based on centrifuging methods which determine the sol's resistance to these magnified gravitational forces.

The object of this work is to determine the relative stability of Bredig gold sols made up within the limiting values of electrolyte and gold concentration as set forth by Miller. Though some of the results seem analogous to those of Briggs it was the object here to use much greater centrifugal forces and vary not only the time but also the speed of centrifuging. This work was done with monovalent ions present which allows higher concentrations of gold to be obtained. The change in potential between the particles and the dispersion medium, due to the change in electrolyte concentration has heretofore been applied only to the sol after it is made up. It is the purpose of the authors to show that this also applies to the sol while it is being made up, thereby correlating the theories explaining both hydrophobic and hydrophilic sols.

Apparatus and Materials

The arcing apparatus and gold wire were the same as was used and described by Beans and Eastlack, excepting that an ammeter and a motor stirrer were added.

All solutions were made up in water having a specific conductivity of 1 to 1.4×10^{-6} mhos. The sodium chloride used was recrystallized three times from conductivity water and dried at 110°C.A standard solution was made up and standardized by evaporating a definite volume to dryness in a weighed platinum dish, dried for several hours in an electric oven at 110°C and gently ignited, cooled and weighed. The standard solution was diluted up to the desired concentration in making up the different solutions used.

The preliminary centrifuging of sols was carried out in a type

three centrifuge of the International Equipment Company. It had rotating arms two feet in diameter and a speed of approximately 2000 r.p.m. giving a force of 1200 times gravity.

A Sharpless laboratory supercentrifuge was used for the final centrifuging of the sols. The bowl or containing vessel, into which the sols were placed had an inside diameter of 2-5/8 inches for the upper half and 1-3/16 inches for the bottom half. When the centrifuge was run the sol was carried up into the larger half of the bowl and there the gold was thrown out. The bottom part was lined with a continuous gold sheet lining. The upper part, having two fins running from top to bottom, could not be so lined but was heavily plated with gold three times and burnished after each plating. This lining was found necessary to prevent precipitation of the gold from the sol by other metals used in the construction of the bowl.

The burettes, pipettes, volumetric flasks and set of weights were standardizes by the usual methods.

Method of Procedure

Sodium chloride was the only electrolyte used in this work. Two hundred c.c. of a solution of this salt of given concentration was placed in a pyrex beaker, a cover placed over the beaker and through holes in this cover the glass stirrer, thermometer and gold wire electrodes were inserted. The glass stirrer was connected to a motor and speed regulating device which allowed a constant rate of stirring while the sol was being prepared. The breaker of solution was surrounded by a bath of ice which allowed the temperature of the sol to be regulated between 16° and 22°C. The arcing was continued for different lengths of time, giving different concentrations of sol, the sol removed and placed in well steamed out tubes and centrifuged for an hour and a half at 1200 times gravity. The sol was then pipetted out, leaving the coarse material in the bottom of the tubes. 25c.c. samples of this sol were centrifuged for different times and at different speeds in the supercentrifuge. For example, 25c.c. were centrifuged for three minutes at 40,000 r.p.m. and 20c.c. pipetted out for analysis. 25c.c. more of the original sol were then centrifuged for seven minutes at 40,000 r.p.m. and 20c.c. pipetted out for analysis. This procedure was carried out for 15, 30 and 60 minute centrifugings. Curves of the type shown in Fig. I, II, III and IV were thus obtained. In like manner the points in the last four figures were obtained by centrifuging separate 25c.c. samples of sol for fifteen minutes at the various speeds shown. To the 20c.c. portions pipetted out for analysis four or five drops of concentrated HCl were added to precipitate out the gold. They were then filtered, ignited and weighed. The gold concentrations given in the following tables are in grams per liter of sol. During the

arcing the current was kept constant at 3.6±.05 amps. The beaker, centrifuge tubes and gold lined bowl were well steamed out after each run.

It was found in centrifuging sols of high gold content in the supercentrifuge that the gold had been thrown out on the walls of the bowl as a black film, and the results of an hour or half hour's centrifuging were higher than those after three, seven or fifteen minutes. It is apparent that these results are in error. Several means of overcoming this were tried and finally it was found that by dividing these two long runs into fifteen minute periods and cleaning out the bowl after each run, this difficulty was overcome and plausible results were obtained.

The speed of the supercentrifuge was determined by a tuning method. To the shaft of an electric motor was attached a metal disk containing three rings of holes, the rings containing 20, 30 and 40 holes respectively. The speed of the motor was determined with the aid of a speed counter and a stop watch. If, for example, the motor was regulated to run at a speed of 1000 r.p.m. and a jet of air was directed against the ring of 40 holes there is a sound of definite pitch given off, corresponding to that given off by a disk containing one hole revolving at a speed of 40,000 r.p.m.. The centrifuge, which was run by compressed air, was started and brought up to a speed which gave the same note as that of the siren. At lower speeds a speed counter attached to the centrifuge, was used to check up on this method of determining the speed of the centrifuge and it was found that this siren method gave the correct speed. This was done at speeds up to 30,000 r.p.m. but was considered too dangerous to the centrifuge to try it at the two higher speeds used.

Check runs were made throughout the work and it was found that at comparatively low concentrations of gold check results were possible but when the sol approached the saturation point, with respect to gold content, such checks were not always possible. In this case the lowest results were used.

Fig. 1

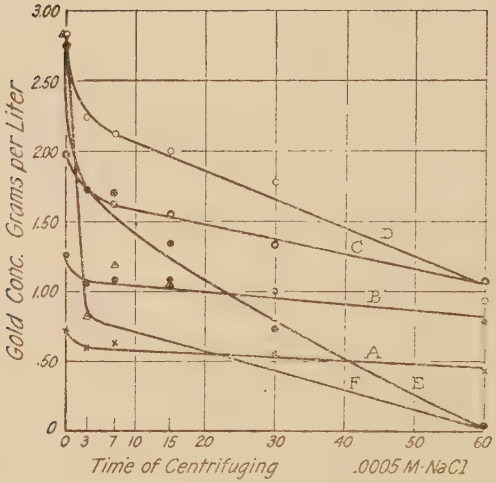

TABLE I

.0005 M Na Cl

grams ler liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	E	F
0	.....	.720	1.260	1.970	2.830	2.745	2.825
3	40000	.595	1.055	1.730	2.245	1.720	.820
7	40000	.630	1.085	1.625	2.125	1.710	1.185
15	40000	.645	1.080	1.545	1.950	1.340	1.040
30	40000	.560	.995	1.325	1.780	.730	.540
60	40000	.425	.790	1.075	.925	.040	.025

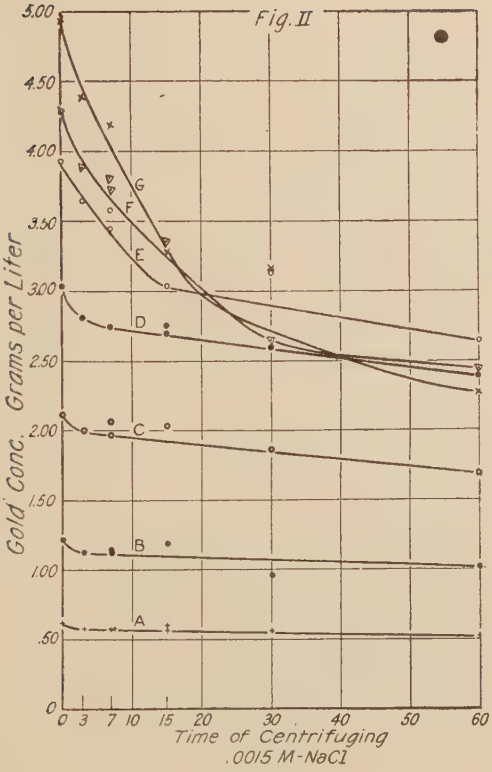

TABLE II

.0015 M Na Cl

grams per liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	E	F	G
0	.....	.615	1.220	2.115	3.030	3.930	4.300	4.940
3	40000	.580	1.130	2.000	2.805	3.645	3.885	4,390
7	40000	.580	1.120	1.975	2.745	3.580	3.815	4.190
15	40000	.570	1.190	2.035	2.705	3.040	3.290	3.285
30	40000	.565	.905	1.865	2.600	3.130	2.640	3.170
60	40000	.525	1.020	1.690	2.395	2.645	2.440	2.275
7	40000	.585	1.150	2.065		3.445	3.725	
15	40000	.600			2.755	3.410	3.365	

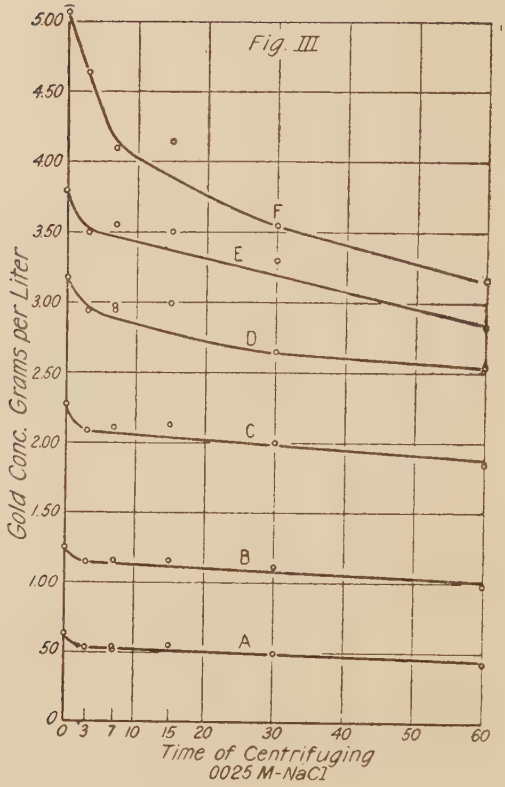

TABLE III

.0025 M Na Cl

grams per liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	E	F
0	.....	.615	1.250	2.270	3.175	3.800	5.065
3	40000	.535	1.150	2.090	2.945	3.500	4.635
7	40000	.525	1.155	2.110	2.985	3.555	4.095
15	40000	.550	1.160	2.125	2.995	3.505	4.145
30	40000	.495	1.115	2.005	2.645	3.295	3.545
60	40000	.420	.985	1.845	2.535	2.830	3.145
7	40000	.545	1.160		2.955		
15	40000	.550	1.170				

Fig. IV

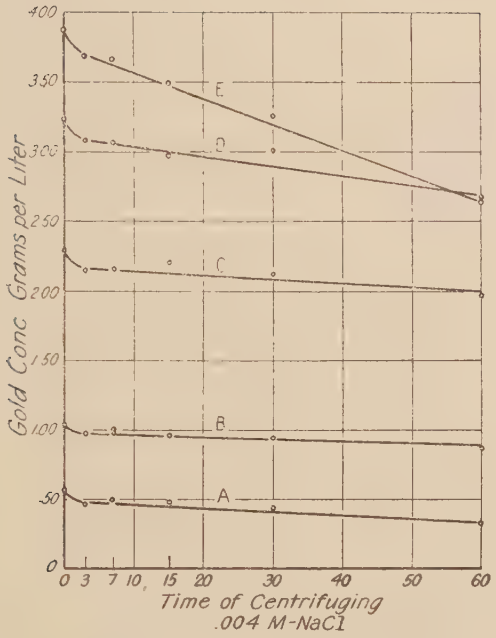

TABLE IV

.004 M Na Cl

grams per liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	D
0	.....	.570	1.030	2.295	3.230	3.875
3	40000	.465	.975	2.150	3.080	3.695
7	40000	.500	.985	2.155	3.070	3.675
15	40000	.485	.965	2.210	2.965	3.495
30	40000	.445	.945	2.120	3.010	3.260
60	40000	.335	.875	1.970	2.680	2.635
7	40000		1.005	2.165	3.080	

Fig. V

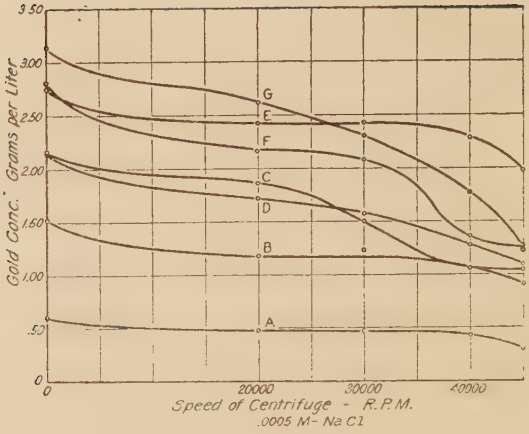

TABLE V

.0005 M Na Cl

grams per liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	E	F	G
0	.....	.600	1.515	2.160	2.140	2.735	2.815	3.130
15	20000	.476	1.178	1.860	1.715	2.420	2.170	2.615
15	30000	.464	1.230	1.500	1.570	2.430	2.080	2.490
15	40000	.426	1.057	1.060	1.275	2.280	1.350	1.770
15	45000	.298	.910	1.045	1.095	1.970	1.265	1.220

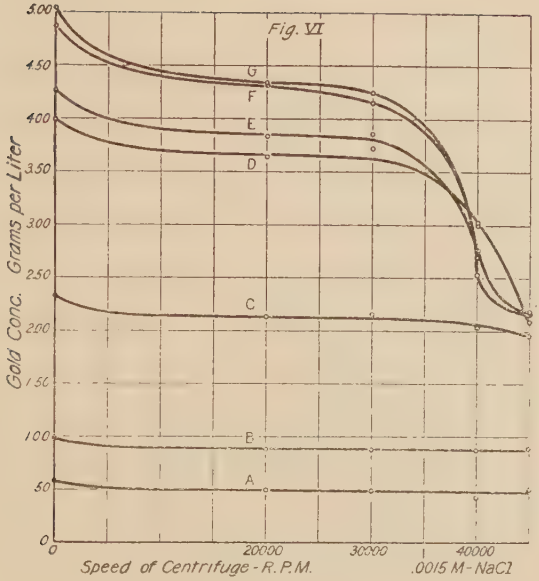

TABLE VI

.0015 M Na Cl

grams per liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	E	F	G
0	.....	.580	.985	2.325	3.990	4.270	4.870	5.035
15	20000	.495	.880	2.130	3.645	3.830	4.315	4.325
15	30000	.490	.880	2.160	3.710	3.865	4.145	4.245
15	40000	.430	.880	2.030	3.025	2.700	2.535	2.765
15	45000	.505	.900	1.965	2.095	2.940	2.165	2.180

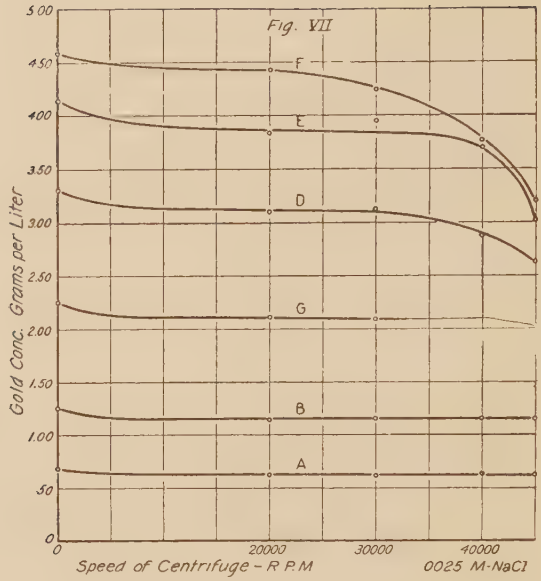

TABLE VII

.0025 M Na Cl

grams per liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	E	F
0	.....	.685	1.260	2.245	3.310	4.140	4.590
15	20000	.635	1.155	2.110	3.105	3.840	4.430
15	30000	.620	1.160	2.095	3.125	3.945	4.340
15	40000	.640	1.160	2.085	2.880	3.700	3.765
15	45000	.635	1.155	2.025	2.630	3.015	3.205

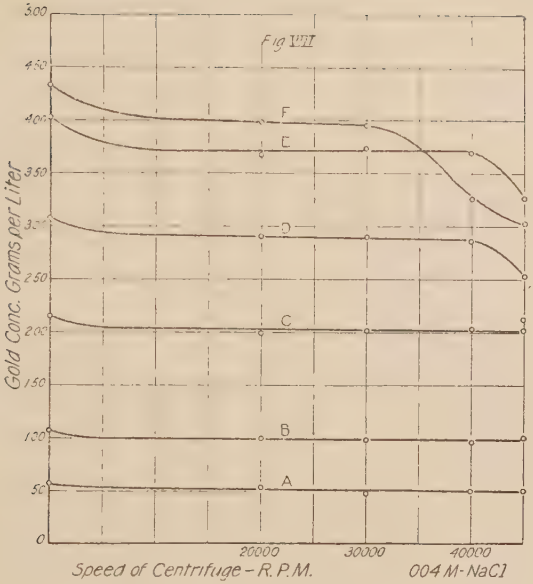

TABLE VIII

.004 M Na Cl

grams per liter of gold after centrifuging

Time in minutes of centrifuging	Speed of centrifuge	A	B	C	D	E	F
0	.....	.565	1.075	2.145	3.075	4.020	4.330
15	20000	.520	1.000	1.995	2.910	3.680	3.975
15	30000	.480	.995	2.020	2.905	3.735	3.960
15	40000	.505	.960	2.035	2.865	3.710	3.270
15	45000	.505	1.005	2.135	2.535	3.270	3.030

These curves picture an increase in stability with a corresponding increase in electrolyte concentration up to a certain point beyond which there seems to be a decrease in stability. They also show the decrease in stability with increasing gold concentration. That this change is not due alone to the size of particles can be seen by comparing the relative positions and shapes of the curves in the different figures. Further proof of the "complex" theory of Bredig gold sols is hardly necessary but some interesting proof is given by these results. It is very evident that if the stability of these sols were due to simply the size of the particles, a sol of about 3 gm per liter, for example, should be as stable when made up in .0005 M Na Cl as in .0015 M Na Cl. The data given here shows that this is not the case for a decided difference can be seen in the properties of the two. (Fig. I & II and Fig. V & VI). Fig. I curve D shows a large decrease in gold content with every increase in time of centrifuging. In Fig. II the curve D with a corresponding original gold content shows a much smaller decrease in gold concentration with increasing time of centrifuging. In Fig. I curve E represents a sol arced beyond the maximum gold concentration and with this sol there is a still greater decrease in gold content reaching practically zero after sixty minutes of centrifuging. This marked decrease in stability is reached in a sol of .0015 M Na Cl concentration by arcing for a much longer time than is given by curve G, Fig. II. The same relative conditions are shown by a comparison of Fig. V and VI where the speed in varied and the time held constant.

The same comparison hold throughout all these sets of curves. Such a comparison of these results, as has just been made, shows the fallacy of considering Brownian movement as the cause of stability.[15] This phenomenon depends upon the size of the particles and as the size of particles is not the determining factor in the stability of sols Brownian movement resolves itself into an effect and not the cause of stability. No attempt was made to cover the stability of sols containing their maximum gold content but the work here follows in the same manner as that of Miller in that the sols of maximum stability are those made up in the concentrations of electrolyte that give a maximum gold content. The results given here are also in accord with those obtained by Briggs for the same kind of results were obtained using a supercentrifuge revolving at a speed of 40,000 r.p.m. as Briggs obtained using a much smaller centrifugal force.

To explain these results satisfactorily it is evidently necessary to accept some theory of complex formation between the gold particles and the stabilizing electrolyte. There must be two opposing tendencies or forces applying themselves to the colloidal particles. We will neglect for the time the effect of the positive

ions in the solution, which tend to precipitate the gold, and consider only the conditions of the particles themselves.

Briggs considers that up to a certain limitating concentration the more electrolyte present, i. e. the more chloride ions present, the more nuclei could be formed which grow, with continued arcing, to particles of larger size, finally reaching a size so great that they are thrown out by centrifuging. A comparison of the curves in Fig. V, VI, VII and VIII would indicate that this idea is correct. It will be noticed that centrifuging sols of lower concentrations throws out very little gold and the curves representing these are straight lines. At a sufficiently high concentration of gold, the straight line curves give place to curves with a sharp break or drop. Starting with Fig. V the first curve shows a slight drop; in Fig. VI the third curve, and in Fig. VII and VIII this sudden decrease in gold concentration does not occur till a sol of higher original gold content is reached, as is shown by the fourth curves in these two latter figures.

According to Kruyt and Van der Spek[16] and as predicted by Wilson, an increase of electrolyte concentration diminishes the potential difference between the colloid particle and the bulk of the dispersion medium, thereby increasing the tendency of the particles to coagulate. Although this theory is offered in explanation of the coagulation of sols by the addition of electrolyte after the sol has been prepared, this idea might be applied to the conditions of a sol during its preparation. From this then it would seem that a sol prepared in .004 M Na Cl having a concentration of gold of .56 gm. per liter (curve A Fig. VIII) for example, would be more easily thrown out than a sol of equal gold concentration in .0015 M Na Cl (curve A Fig. VI) for the former has a larger excess of electrolyte present. According to the results shown such is not the case for the two curves mentioned are practically identical in form. The sols made up in more concentrated electrolyte solutions are not less stable than those made up in lower electrolyte concentration. In fact there is an increase of stability with increase of electrolyte concentration up to a certain limit.

J. A. Wilson[10] has applied the Donnan membrane equilibrium theory to that of the "complex" theory of colloids. He pictures a colloidal particle of gold prepared in a solution of an electrolyte M N. The negative ion N^- is combined or joined to the particle by electro chemical attraction, which is due to t he residual valence of the gold particle. In the liquid layer imediately surrounding the particle there are M^+ ions which are held in this layer by the negative charges of the now "complex" colloid particle. There will be, therefore, a different distribution of ions in this layer than in the bulk of the solution, resulting in a condition to which the

Donnan membrane equilibrium theory can be applied. This surface layer will contain also M^+ and N^- ions. The product of (M^+) and (N^-) will be equal in the surface layer and bulk of solution acording to the Donnan theory. However the concentration of M^+ and N^- are unequal in the surface layer but equal in the bulk of the solution. The total concentration of ions in the surface layer will be greater than in the bulk of solution. This difference in distribution of ions gives rise to a difference in potential between the surface layer and the bulk of the solution. The amount of electrolyte which can combine with a colloid of definite degree of dispersion has a limited maxium value, due to the amount of residual valence on the surface of the particle. According to this then, the excess electrolyte present when the sol is first being prepared does not lessen the stability of the sol. Not till after the combination or ratio of colloid to electrolyte has reached or neared its maximum has any excess electrolyte the effect of lowering the potential difference and allowing the particles to coagulate.

As shown in Fig. V, VI, VII, VIII up to a certain gold concentration the sols are very stable. Above that ratio of gold and electrolyte the curves make sharp breaks and the higher the concentration of gold the more easily the gold is thrown out. Briggs has shown that there must be a maximum number of particles possible for each concentration of electrolyte and after that maximum is reached any increase in gold content must be due to the growth of these particles. This is very apparent also from the results given here. Before the gold electrolyte maximum combination has been reached the increase in size of the particles has little or no effect compared with the potential that holds them in a stable condition. After reaching this maximum combination however, the increase in size does play an important part, causing the gold to be more easily centrifugated out. This effect increases with increasing original gold content of sol and when saturation with respect to gold is reached (as outlined by Miller) there is a very rapid decrease in gold concentration with either increase in speed or time of centrifuging.

This theory of colloid stability, as outlined by Wilson, led the author to try two or three experiments in dilution of sols. If increasing the concentration of electrolyte in a sol decreases the potential difference and thereby allows the particles to coagulate more rapidly, then decreasing the electrolyte concentration should increase this potential and make them more stable. Thomas and Foster found this to be the case with tannin.[17]

60cc of the sol given in table VIII F (Au conc. of 4.33 gm/l) was diluted to twice it's volume with conductivity water. The results are given in the following table.

TABLE IX

Time in minutes of centrifuging	Speed of centrifuge	gm of gold in 20c.c. of sol after centrifuging	gm liter of gold after centrifuging
0	.....	.0433	2.665
15	20000	.0409	2.045
15	30000	.0398	1.990
15	40000	.0397	1.985
15	45000	.0397	1.985

There was a slight decrease after centrifuging at 20,000 r.p.m. but no further marked decrease at the other speeds, as was the case with the original sol before dilution. Either the large particles of the sol are broken up by the dilution or the potential difference is increased causing the sol to become more stable. The latter explanation seems to be the more plausable one.

A second such test was made and the results are given in the following table .50 c.c. of the sol given in table II C were diluted to 150cc with conductivity water resulting in a sol of 1.64 gm Au per liter and an electrolyte concentration of .0005 M Na Cl.

TABLE X

Time in minutes of centrifuging	Speed of centrifuge	gm of gold in 20c.c. of sol after centrifuging	gm liter of gold after centrifuging
0	.....	.0328	1.640
15	20000	.0311	1.555
15	30000	.0306	1.530
15	40000	.0271	1.355
15	45000	.0245	1.225

There is a slight decrease in gold concentration at 40,000 and 45,000 r.p.m. over that at either 20,000 or 30,000 r.p.m. but the results follw those which would be obtained by arcing a sol to this gold concentration in .0005 M Na Cl.

SUMMARY

1. Further evidence is given in suport of the complex theory of colloid formation.

2. More definite means of determining stability of Bredig gold sols have been given by the centrifuging method.

3. The stability of Bredig gold sols increases with increase in electrolyte concentration up to a certain value then decreases. For a definite concentration of electrolyte the stability of the sol decreases with increase concentration of gold after a certain gold electrolyte ratio has been reached.

4. With any particular electrolyte there is a maximum ratio of gold to stabilizing electrolyte, above which an increase in gold concentration gives a marked decrease in stability as determined by the centrifuging method.

5. There is a further marked decrease in stability of sols arced beyond the saturation point for gold.

6. The stability of Bredig sols is not due primarily to the size of the particles but to the conditions under which the sol is made up.

7. A preliminary application of the Donnan membrane theory as outlined by Wilson has been made in explanation of the results obtained in this work. As far as the author has been able to ascertain this is the first experimental work given to show the application of this theory to the formation and behavior of Bredig gold sols. Further work using different methods must be done to prove the validity of this application.

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